भारतीय मानक
Indian Standard

IS 14507 : 2023

डाइफिनाइल ईथर — विशिष्टि

(पहला पुनरीक्षण)

Diphenyl Ether — Specification

(First Revision)

ICS 71.100.60

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

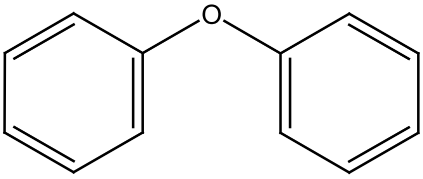
April 2023 **Price Group 6**

Fragrance and Flavour Sectional Committee, PCD 18

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Diphenyl ether or Diphenyl oxide does not occur in nature. Due to its low cost and stability it is extensively used in various types of fragrances for detergents, soaps and many other household and industrial products for its rose and geranium like odour effect. In conjunction with diphenyl, it is extensively used as a high temperature heat transfer medium (Dowtherm). It is represented by the following structural formula:



Structure of Diphenyl ether (Diphenyl oxide)

(Molecular Weight 170.21)

It is also known as phenyl ether or diphenyl oxide or phenoxybenzene. In India, diphenyl oxide is produced commercially mainly by two methods. The main method is by heating alkali phenolate with chlorobenzene at elevated temperature in the presence of a catalyst or by passing phenol over a catalyst at 400 °C.

This standard was first published in 1998. In this (*first revision*), the gas chromatographic analysis for purity determination of *p*-cresyl methyl ether has been upgraded from Packed Column GC to Capillary Column GC for more accurate results. Peroxide value limit has been incorporated in this revision.

The composition of the committee, responsible for the formulation of this standard is listed in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test of analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value shall be the same as that of the specified value in the standard.

Indian Standard

DIPHENYL ETHER — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for diphenyl ether.

2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed in Annex A.

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597 shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 The material shall be a clear, nearly colourless liquid, solidifying at or below room temperature. The material shall be free from phenol, moisture and free chlorine and other extraneous matter and impurities.

4.1.2 The material shall also be tested olfactorily and especially for objectionable/undesirable by odours as prescribed under IS 2284.

4.2 Solubility

The material shall be clearly soluble in **5** parts of ethanol (90 percent by volume) when tested as prescribed under IS 326 (Part 6).

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminium, as agreed to between the purchaser and the supplier. The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Batch number and date of manufacture;
d) Net and gross mass;
e) Net Volume of the material; and
f) BIS Certification Marking.

The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian *Standards Act, 2016* and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 Number of Tests

All the characteristics given under **4** shall be tested on the composite sample.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under **4.1** to **4.3**. Reference to relevant Indian Standards and Annex is given in co1 (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Diphenyl Ether
(*Clauses* 4.3 and 7.1)

Sl No.	Characteristic	Requirement	Requirement Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Odour	Strong 'Geranium leaf like odour, free from phenolicand chloro compounds off-odours	IS 2284
ii)	Relative density at 27 °C	1.071 to 1.074	IS 326 (Part 3)
iii)	Refractive index at 27 °C	1.577 2 to 1.578 0	IS 326 (Part 5)
iv)	Congealing point, *Min, °C*	26.7	IS 326 (Part 9)
v)	Freedom from chlorine	Shall pass the test	Annex B
vi)	Freedom from phenol	Shall pass the test	Annex C
vii)	Peroxide value, *Max*	3.0	Annex D
viii)	Determination of Purity, percent by mass, *Min*	99	Annex E

NOTE — The correction factor for Relative Density and Refractive Index for each degree celsius change in temperature are 0.000 64 and 0.000 38 respectively.

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 2022	Sampling (*fourth revision*)
(Part 3) : 2006	Determination of Relative density (*third revision*)
(Part 5) : 2006	Determination of refractive index (*third revision*)
(Part 6) : 2005	Evaluation of miscibility in ethanol (*third revision*)
(Part 9/Sec 2) : 2017	Determination of ester value and free alcohols, Section 2 Estimation of free alcohols content by determination of ester value after acetylation (*third revision*)
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 2284 : 1988	Methods of olfactory assessment of natural and synthetic perfumery materials (*first revision*)
IS 6597 : 2001	Glossary of terms relating to natural and synthetic perfumery materials (*second revision*)

ANNEX B

[*Clause* 4.3, *Table* 1, *Sl No.*(v)]

TEST FOR FREEDOM FROM CHLORINE

B-l OUTLINE OF THE METHOD

A copper gauge is ignited in the flame with few drops of the sample, if the material is free from halogen, the flame shall show no green colour.

B-2 APPARATUS

B-2.1 Copper Gauge — Strip of 20 mesh and 1.5 cm × 5 cm

B-2.2 Copper Wire

B-2.3 Bunsen Burner

B-3 PROCEDURE

Wind a strip of 20 mesh copper gauge 1.5 cm long around the end of a copper wire. Heat the gauge in a non-luminous flame of the Bunsen burner until it glows without colouring the flame green. Permit the gauge to cool and re-heat several times until a good coat of oxide has formed. Apply with a medicine dropper, 2 drops of sample to the cooled gauge, ignite, and permit it to burn freely in the air. Again cool the gauge and add 2 more drops of the sample and burn as before. This process is continued until a total of 6 drops has been added and ignited. Then hold the gauge in the outer edge of the Bunsen flame, adjusted to a height of about 4 cm. If the material is free from halogen, the flame should show no green colour.

ANNEX C

[*Clause* 4.3, *Table* 1, *Sl No.* (vi)]

TEST FOR FREEDOM FROM PHENOL

C-l OUTLINE OF THE METHOD

The aqueous filterate of the material is treated with ferric chloride solution, a greenish, purplish or bluish colour indicates the presence of phenol or cresol.

C-2 REAGENTS

C-2.1 Ferric Chloride Solution — 10 percent

C-3 PROCEDURE

Shake vigorously 1 ml of the sample with 10 ml to 20 ml of distilled water in a glass-stoppered test tube. Allow the liquids to separate and filter through filter paper moistened with distilled water. To the aqueous filtrate. Collected in a test tube, add 2 drops to 3 drops of a 10 percent solution of ferric chloride. A greenish, purplish or bluish colour in indicates the presence of cresol or phenol.

ANNEX D

[*Clause* 4.3, *Table* 1, *Sl No.* (vii)]

DETERMINATION OF PEROXIDE VALUE

D-1 REAGENTS

D-1.1 Acetic Acid — Analytical reagent grade

D-1.2 Chloroform

D-1.3 Sodium Thiosulphate Solution — 0.01 N approximately

D-1.4 Potassium Iodide — saturated solution

D-1.5 Starch — freshly prepared saturated solution

D-2 PROCEDURE

D-2.1 Weigh accurately about 2 g of the material into a conical flask (250 ml capacity) with provision for stoppering. Add 25 ml of acetic acid chloroform mixture solution (3 : 2 *v/v*). Swirl to dissolve the material completely. Add 2 ml of freshly prepared saturated aqueous solution of potassium iodide. Allow the solution to stand with occasional shaking for 1 min and then add 35 ml of distilled water. Add 2 ml of freshly prepared starch solution. Titrate against sodium thiosulphate (0.01 N). End point being the disappearance of the blue colour. A blank titration using all the reagents but without sample should be carried out.

D-3 CALCULATION

D-3.1 Calculate the peroxide value in the material as follows:

$$\text{Peroxide value, milli equivalents/kg} = \frac{(V_1 - V_2) \times N \times 1\,000}{M}$$

where

$V1$ = volume, in ml, of sodium thiosulphate solution used for the test sample;
$V2$ = volume, in ml, of sodium thiosulphate solution used for blank titration;
N = normality of sodium thiosulphate; and
M = mass, in g, of the test sample.

ANNEX E

[*Clause* 4.3, *Table* 1, *Sl No*. (viii)]

DETERMINATION OF PURITY

E-1 OUTLINE OF THE METHOD

Equal weight of samples (diphenyl ether; 5mg) and internal standard (nonane; 5mg) were dissolved in dichloromethane (1 ml) and injected in a gas chromatograph fitted with autoinjector, where the carrier gas (nitrogen) from one end of the column to the other carried it. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column. The detector signals on transmission to the recorder plots the chromatogram.

E-2 APPARATUS

Gas chromatography (GC) (Shimadzu GC 2010 system equipped with an FID) was applied for the identification of individual components. The typical chromatogram for diphenyl ether (Ultra 1, Ultra 2 and KDAC sample) was presented in Fig. 1, 2 and 3. The chromatographic conditions were mentioned as below:

Sample	Diphenyl ether
Column Material	Capillary column (SH-Rxi-5Sil MS) Similar to 5% diphenyl/ 95% dimethyl polysiloxane (equivalent to DB-5, HP-5)
Length	30 m
I.D.	0.25 mm ID
Film thickness	0.25 µm
Carrier gas	Nitrogen
Column temperature	70 °C for 5 min and then continuously increased to 220 °C @ of 7 °C/min and held for 5 min
Injector port temperature	240 °C
Injection volume	2 ul
Mode	Split
Split ratio	10 : 1
Pressure	87.4 kPa
Column flow rate	1.05 ml/min
Detector type	FID
Detector Temperature	260 °C
KI (Kovats indices) value	
Literature, 1396	Calculated ~1410
AI (Arithmetic index) value	Calculated ~1409

NOTE — The above gas chromatographic conditions are suggestive /typical. However, any GC with equivalent column may be used provided standardization/calibration is done after setting up chromatographic conditions for the desired/required resolution.

E-3 PROCEDURE

The gas chromatography of diphenyl ether was done under the condition indicated in **D-2**. After stabilization of the desired temperature of the column, injection port and detector, 2 µl of the sample was injected. If required, sample is re-injected to get better chromatogram.

E-4 CALCULATION

Percentage area for components was automatically calculated by the software of the used machine and was found in the ratio around Nonane : Diphenyl ether (46.5 : 53.5).

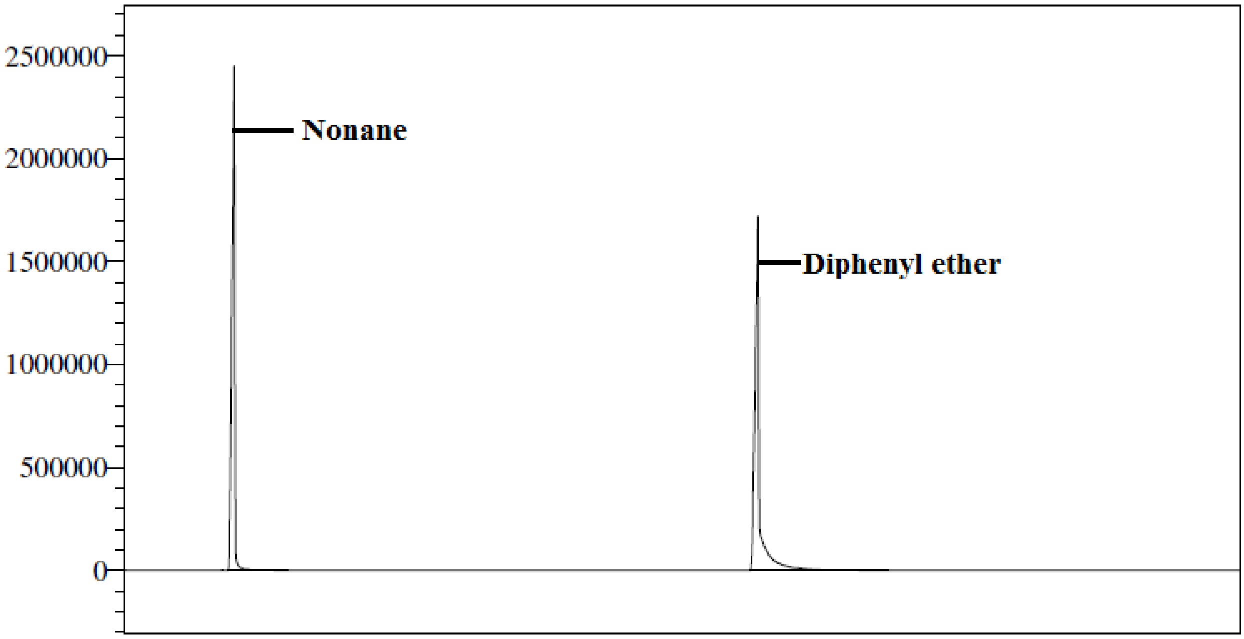


FIG. 1 A TYPICAL CHROMATOGRAM OF DIPHENYL ETHER

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Fragrance and Flavour Sectional Committee, PCD 18

Organization	*Representative*(*s*)
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Aroma Sales Corporation, New Delhi	Shri Sunil Kumar Jain
Central Drugs Standard Control Organization, New Delhi	Shri Aseem Sahu
Central Drugs Testing Laboratory, Chennai	Shrimati C. Vijayalakshmi
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Consumer Voice, New Delhi	Shri B. K. Mukhopadhyay
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In Personal Capacity (*Flat No. 201,Vishakha CHS Ltd, off, Swami Netyanand Marg, Near Gokhale Bridge, Andheri East - 400069*)	Shri G. S. Ranade
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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor,
DMRC Building, Bhavbhuti Marg, New
Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V,
Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg,
Chandigarh 160019 — 265 9930

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